Final Performance Report

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Final Report for Fiscal Year 2007 DURIP Grant

Summary: A research grade Dynamic Mechanical Analyzer (DMA) was acquired in 2007 through a successful Department of Defense grant under the DURIP program. This equipment has played a key role in the evaluation of candidate polymeric materials for developing reconfigurable, or morphing, aerospace structures. In particular, shape memory polymers (SMP) in filled and unfilled form have been investigated with particular emphasis on the recovery time and force as the materials Response time and recovery force are performance undergo transformation. characteristics essential to the design of SMP based actuators and reconfigurable structures, and new testing protocols have been developed to quantify these merits of performance. While favorable response times have been observed, the low recovery force measured in experiments, and the bulky size of the triggering mechanism have presented themselves as challenges in the development of efficient and lightweight structures. However, the provision of the equipment is enabling more research into the modification of the polymer's properties to overcome these shortcomings. Ongoing approaches include alternation of the SMP's chemistry and the use of different fillers such as carbon black, aniline, and micron sized iron and copper particles.

Experimentation

<u>Equipment Description</u>: The DMA, which is a model RSA3 manufactured by TA Instruments, is shown in Fig. 1. The DMA is a high precision, low load dynamical testing apparatus and is equipped with TA's full range of testing accessories enabling tensile, compressive, shear, and three point bending tests, the last being a configuration that is well suited for high stiffness



Figure 1. Illustration of DMA with liquid nitrogen cooling unit installed in the Polymer Research lab at Miami University.

materials. This apparatus can also perform high frequency measurements on films and fibers. An integral environmental chamber allows the determination of the effect of temperature on mechanical properties of the test alongside material. measurement of viscoelastic effects through changes in the applied loading rate. The environmental chamber is critical for replicating service operating temperatures of a SMP based aerospace part and for determining triggering the temperature of SMPs. Various specifications of the machine are

provided in Table 1.

Standard material properties determinable through the aforementioned test configurations include, glass transition temperature, storage and loss modulus, creep compliance, relaxation modulus, and viscoelasticity range.

Table 1. Key specifications of the RSA3 dynamic mechanical analyzer.

Parameter	Capacity			
Maximum force	35 N			
Force resolution	0.0001 N			
Strain resolution	1 nm			
Dynamic strain deformation range	± 0.5 to 1500 μm			
Frequency range	2×10 ⁻⁵ to 80 Hz			
Modulus precision	1%			
Temperature range	-150 to 600 °C			
Heating and cooling rate	0.1 to 60 °C/min			

<u>Test Procedure:</u> In addition to these basic tests, a special test program has been prepared to allow a measurement of the recovery force and response time. This is a two stage process. The first stage entails, 1) elevating the temperature of the sample to the trigger point, 2) a dwell time interval (to allow for heat soak), 3) deformation of the sample to a set strain, and 4) cooling the sample to room temperature while keeping the strain locked-in. This stage results in a sample set into a 'reconfigured' state. On the DMA, this test was performed in the three point bending mode. The results from the bending test have been juxtaposed against tensile test data using dog-bone shaped specimen tested in an Intron servo-mechanical tensile tester with good agreement in the data.

The second stage is aimed at observing the recovery behavior which would mimic the action of a SMP based actuator or flight surface for instance. In this stage, the deformed sample is re-triggered (re-heated in this case) while the machine is set to prevent strain recovery in the sample. Force and time data is recorded.

Discussion

Development of triggering mechanism: In a SMP, the glass transformation temperature typically coincides with the triggering temperature. The DMA's temperature sweep capability has been used to determine this value for each of the two shape memory polymers: the styrene based Veriflex, and the new epoxy based Veriflex-E. The measured values are 75 °C and 100 °C, respectively. This test was repeated for iron filled Veriflex samples (10% and 20% by mass loading of iron particles less than 10 microns). No change in the glass transition temperature was observed, and subsequent mechanical shear and tensile testing revealed little change in the elastic modulus and tensile strength of the filled versus the unfilled samples. This implies an absence of molecular level interaction between the chains and the filler particles.

Preliminary tests performed at the Wright Patterson AFB, Dayton, OH, Hybrid Materials Branch, showed inductive heating as a promising triggering mechanism, which spurred investigation into the use of ferromagnetic filler particles to generate uniform through-body heat in an SMP to ensure rapid triggering. A line of investigation in this research project has been to confer electrical conductivity to the SMP in order to use ohmic heating to circumvent the need for a bulky induction heating system. However, initial trials with fillers including carbon black, and micron sized iron particles have not been successful. The standard Veriflex resin has been found to not cure well with loading percentages higher than 20%. The new Veriflex-E, also produced by Cornerstone Research Group of Dayton, is expected to address this shortcoming and is currently being tested by the PI.

<u>Testing results:</u> Two merits of performance essential to the successful development of SMP based morphing structures, whether they constitute the skin or an auxetic style sub-structure of an aerodynamic element, have been identified, and they are response time and recovery force. The experimental details of how these characteristics are measured have been presented in 'Experimentation' section. The DMA acquired modulus versus temperature data is presented in Fig. 2 for 20% wt Fe filled Veriflex This plot is nearly indistinguishable from that of an unfilled Veriflex specimen vis-à-vis modulus and glass transition temperature, thereby alluding to lack of filler-chain interaction.

The DMA derived force-time response curves are shown in Figs. 3 and 4 for Veriflex and Veriflex-E. With both materials, the almost instant 'on' nature of the material's recovery process when the trigger temperature is reached is seen in the graphs in the steep slope of the force-time plots for an initial near-zero force. While this result bolsters the case for the use of SMPs in a time sensitive application, these positive findings are somewhat mitigated by the low measured loading bearing/application capacity of the SMPs as they revert to their original state. The recovery force, a quantitative assessment of the materials propensity to revert to its original state, has been measured to be approximately 12-15% of the flow stress in the softened state, which itself is only approximately 15% of the room temperature tensile strength. Therefore, this means

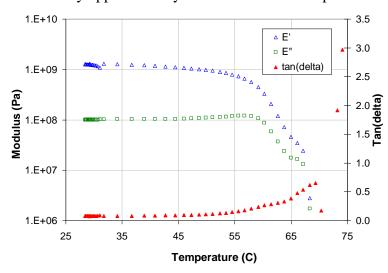
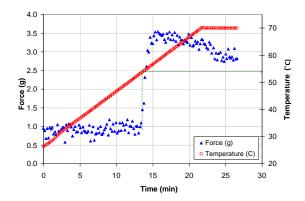


Figure 2. DMA data from 1Hz dynamic loading of a filled Veriflex sample.

an actuator or any other SMP based part can sustain only a fraction of its load bearing capacity as it undergoes transformation. This aspect of SMP behavior is critical to the design of SMP based components.



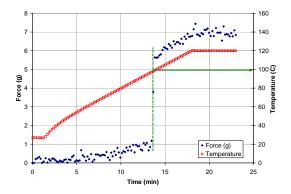


Figure 3. Recovery force and response time data for unfilled Veriflex SMP.

Figure 4. Recovery force and response time data for unfilled Veriflex-E SMP.

Applications of interest to the military

The development of morphing structures, whether it be through the use of a SMP or another material, can greatly benefit the performance and functionality of aero vehicles. The materials characterization research presented in this report constitutes a critical component in the feasibility study of SMPs. While the time response of a SMP seems amenable to the development of a viable reconfigurable structure, the recovery force of the SMPs is very limited, and, therefore, this shortcoming must be compensated for through, perhaps, and freely articulating support structure. To that end, future research using the new and existing equipment is directed at,

- 1) Improving the stiffness of the materials in the softened state through the use of a filler that partially restricts large segmental motion of the polymer chains. However, an undesirable side effect might be restriction of the deformed strain, and so this path might have to be followed in conjunction with the development of a freely articulating support structure to support external loads.
- 2) While initial trials with spherical iron particles (<10 microns) and carbon black have been unsuccessful in conferring electrical conductivity to the resin, two new fillers are being investigated. Since joule heating through the passage of direct current is still considered to be more feasible than inductive heating primarily due to packaging and efficiency advantages, alternative means of achieving this goal may be possible through the use of a highly flexible conductive media in the form of a weave which will be cured into the resin in the form of a laminate.
- 3) Development of a prototype reconfigurable structure following the resolution of the triggering challenge is highly desirable. This would constitute a new enabling technology for lightweight unmanned aerial vehicles.